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RESEARCH ARTICLE

Effect of Liquid Contamination on Hermeticity and Seal Strength of Flexible Pouches with LLDPE Sealant

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ABSTRACT

Flexible packaging is a growing successful market and the majority of flexible package applications are for the food industry. The demand for process optimization and reduced production costs, has led to an increase in flexible packaging. However, fast production lines can result in contamination in the seal area. For flexible food packaging, contamination is considered any food particle or substance trapped in the seal area. Current quality control processes can detect contamination in the seal area, but it is not determined if seal contamination affects seal quality. Oil-based and sodium based snack foods are two common categories that can be packaged on a horizontal form fill and seal (HFFS) flow-wrap machine. The study uses vegetable oil and a salt water solution to simulate the effect of liquid contamination along the T-point of flexible pouches made on an HFFS. The T-point refers to where the fin seal meets the end seal and requires the seal jaw to seal through four layers of film, which is the most difficult point to seal. The study tests a combination of different sealing temperatures and dwell time to determine the optimal sealing condition for a hermetic seal. A quality hermetic seal provides an enclosed seal with no leaks due to successful polymer chain entanglement between the two sealant layers. The different test categories of the study are non-contaminated (control), salt water solution for salt based foods, and vegetable oil for oil-based foods. Given the test parameters of the study, 1400C sealing temperature and 0.3 seconds dwell time are considered to be the optimal sealing condition for all three test categories. For Phase 1 of the study, salt water had a lower Hermeticity pass rate compared to vegetable oil and non-contaminated seals. In addition, the effect of refrigerated storage temperature and ambient storage temperature did not show to be significant for any of the test categories. However, refrigerated conditions showed a higher

Hermeticity pass rate, but it was not statistically different. The findings for seal strength indicated no test category had higher or lower seal strength over the 14 day test period. Overall, the study shows there is no effect of liquid contaminant on Hermeticity and seal strength for flexible film with LLDPE sealant layer.

KEY WORDS*: Flexible Pouch, LLDPE, Sealant, Horizontal Form Fill Seal, HFFS*

1.0 INTRODUCTION

The demand for high production volume requires fast production lines, and especially for flexible food packaging it is common to find food particles trapped in the seal area. Any food particle or substance found in the seal area of a flexible package is considered a contaminant. Quality control processes detect contamination and in some cases the package is discarded due to the assumption that the seal quality is compromised. This study is important to determine the effect of liquid contamination in the seal area for the flexible food packaging industry. As of June 2013, the flexible packaging industry grossed \$26.7 billion dollars in sales with 58 percent for the food packaging industry [1]. Flexible packaging uses less material weight and has the ability to optimize production. According to the Flexible Packaging Association, over a six year time frame, the packaging weight of a candy bar has reduced by 60 percent [1]. The ongoing successful research and development of advanced materials for specialty films gives flexible packaging a strong advantage among other packaging options. A wide range of different film structures offers solutions to prolonging shelf life and other package performance concerns such as contamination. In the food industry, two primary packaging functions are protecting the product from outside contamination and containing the product within the package. The demand for flexible packaging comes from the demand for low cost and high volume production capability. In comparison to rigid packaging, flexible packaging reduces packaging material weight per package. Thinner and lighter weight material can save costs for companies without compromising their packaging needs as well as reducing the environmental footprint. The switch to more flexible packaging requires a trial and error process to determine the optimal temperature and dwell time combination. Moreover, choosing the highest sealing temperature and dwell time is not the most effective option because it can slow down production and can affect seal properties. For flexible packaging, the film chosen for an automated packaging production will have an optimal or range of packaging conditions. In this study, nine different packaging conditions will be analyzed to compare the performance of seal through liquid contamination for oil-based and sodium-based food products.

1.1 Quality Control

Currently, quality inspection for food production inspects for food particles in the seal area among other quality issues. Food particles found in the seal area are considered a contaminant and in some cases lead to leaking. The food particles in the seal area can be aesthetically displeasing to the consumer, and effect the consumer's perception of the product. It is time consuming to inspect every bag manually for contamination, so automated quality control processes were developed to efficiently find packaging defects. Polarized Light is one procedure used in industry to find food particles in the seal area. It is a non-destructive process that uses linear polarized light to pass through transparent film, which shows a color stress pattern once the light passes through the second light filter. Laser scattering imaging is another non-destructive process that measures the light that is deflected from the contaminant found in the seal area [2]. Both types of technologies produce images to inspect for food particles or other contaminants such as metal content in the seal area. In a study completed by Barnes et. al [2], polarized light and laser scatter technology had an accuracy of 96% and 90%. Overall, polarized light and laser scatter technology identify defects in the seal but cannot determine if the defect has an effect on the hermetic seal. A hermetic seal provides a complete enclosed package with no leaks or holes. In addition to food contamination, wrinkles from film overlapping in the seal can also lead to poor seal integrity. These issues can be visibly seen during inspection and detected through automated quality control processes. In this study, liquid contaminants are forced into the seal area to test Hermeticity and seal strength. The quality of the seal is determined by seal integrity, which includes seal strength properties and Hermeticity. Seal strength is the amount of force required to separate the film progressively over time [3]. It is also an important factor for containing the product from the time it is packed to the time it is consumed. However, too high of a seal strength can make it difficult for consumers to open the package.

1.2 Heat Sealing Technology

Heat sealing is commonly used in the flexible packaging industry and includes jaw-type seal bars, rotary sealers, band rotary sealers, bead sealers, hot knife or side-weld sealers [4]. The study uses heat sealing technology using jaw-type seal bars for a horizontal form, fill, and seal flow-wrap machine. There are three parameters for heat sealing: 1) sealing temperature 2) dwell time 3) and pressure. The temperature is an important factor for the sealant surface to reach its molten or partially molten stage. Secondly, the dwell time is the duration the seal jaws come into contact with the film. In this study, the dwell time is considered the actual time period the seal jaws are in direct contact with the film versus the total time the seal jaws are in motion to make each seal. Dwell time allows for the polymer chains to reach molten or partially molten stage to entangle and create a hermetic seal. If dwell time is too short for the polymer chains to reach molten or partial molten stage, the corners and the T-point will have a weak seal and are more likely to show leaks during Hermeticity testing. The T-point refers to the point on the seal where the fin seal meets the end seal. The pressure applied to seal both sides of the film together will remain the same throughout the study. Pressure is needed to seal two film surfaces together, but increasing the pressure has no effect on seal strength [4,5].

The seal jaw temperature is a primary factor for seal properties but the interface temperature is the actual temperature of the sealed surface during the sealing process. Interfaced temperatures are important to reaching desirable sealing properties. This study did not record the interface temperature but monitored the actual sealing temperature of the machine. Future work can include determining the relationship between the set sealing temperature and interface temperature.

According to Meka and Stehling [4], the interfacial temperature is a lower value than the platen temperature. The study also tested the relationship between dwell time and platen temperature. At 130^{o}C, an increase of only 10% interface temperature was observed from 0.4 seconds to 1.4 seconds dwell time. In addition, Meka and Stehling determined the effect of dwell time has less an effect on interface temperature as the sealing temperature increases. Moreover, sealing temperature has more of an effect on seal strength than dwell time.

Moreover, seal jaw styles can differ between machines and different sealing technologies. Matthews et. al [6], studied seal strength and the effect of crimp angle and pitch of the seal jaw for heat sealing processes. The study compared Cellulose (38µm) and PLA (35µm) to OPP (25µm, 35µm, and 50µm) and found that crimp angle is a secondary factor to seal strength. Moreover, the crimp styles with more than 80^{0} angles provide greater seal strength for films outside 25-45 µm. The film used in this study is 65 µm, and crimp style seals were used in the study. The crimp angles of the seal jaws were not determined in this study, but can be determined in future studies. Furthermore, the study showed crimp geometry as a secondary factor behind seal temperature.

Although sealing temperature is one of the two primary factors to reach Hermeticity, it is important to consider the peel force required to open the package. The temperature and dwell time combination may provide the strongest seal strength but it may make it impossible for the consumer to open the package. Companies can increase sealing temperatures with shorter dwell times to expedite the filling process. However, the change in temperature and dwell time to reach the desired interface temperature more quickly can change the seal properties [7].

1.3 Seal Strength

Testing the seal strength determines the amount of force or stress on the seal with respect to the elongation or strain to reach material or peel failure. In this study, the seal strength will be tested during Phase 2 after the optimal sealing condition is determined from Phase 1. Testing the seal strength of a flexible pouch determines the type of seal failure for the given sealing condition.

Figure 1 shows that if the seal bar temperature is above the melting point, T_m, of the sealant, then the seal strength test will show a tearing mode failure.

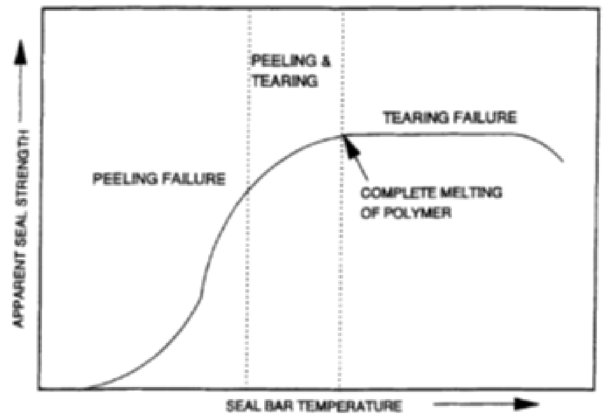

Figure 1: Relationship between Seal Bar Temperature and Apparent Seal Strength for semicrystalline polymer films [4]

On the other hand, the seal strength test will show a peel failure if the seal bar temperature is well below the T_m of the sealant. However, if the seal bar temperature is within close range of the T_m but below the melting point, the seal strength test will more likely result in a peeling and tearing mode failure.

There are several types of results from a seal strength test: peel failure, tear failure, peel and tear failure, and elongation failure (Figure 2). A weld seal will result in a tear failure, which shows that the strength of the seal is stronger than the strength of the film [7]. In addition, there is also delamination failure mode that can occur in combination with the other failure types. Delamination occurs when one of the layers separates from the film during seal strength while either the outer layer or sealant layer remains attached during tensile testing.

According to Yuan et. al [5], a sealing temperature of a few degrees before the melting temperature, T_m, seal strength will significantly increase and result in a peel, delaminating a tear mode or combination of the failure modes. If the sealing temperature is more than a few degrees below the melting point, then a peel failure mode will more likely occur and the result will be a lower strength

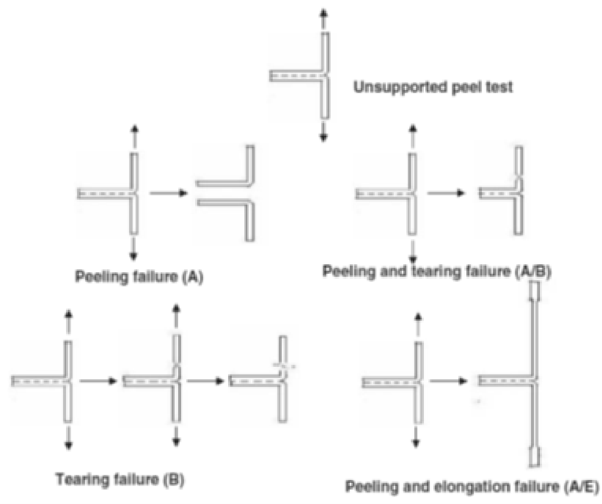

Figure 2: Previous Seal Strength Results found in study completed by [7]

than the other failure modes. Yuan et. al [5] tested a similar structure to what is used in this study, Polyethylene-Terephthalate, PET (film laminate) and linear low-density polyethylene, LLDPE (sealant layer). An increase in seal strength occurred from 0.1 seconds to 1 second dwell time for the majority of sealing temperatures within the range of 103^{o}C and 130^{o}C. Furthermore, the same seal strength can be made at different sealing conditions. For example, a 115^{o}C and 0.2 seconds sealing condition showed the same results for a sealing temperature equal or greater than 118^{o}C with 0.1 seconds.

Tetsuya et. al [8] studied OPP and CPP seal strength at different sealing temperatures and concluded that an increase in temperature showed an increase in material failure at edge of seal. The lower range sealing temperature of 115^{o}C showed more immediate material failure compared to 170^{o}C and 250^{o}C that showed more necking before failure.

1.4 Liquid Contaminants in the Seal Area

A previous study completed by Mihindukulasuriya and Lim [9] investigated seal strength with contamination in the seal area. According to Mihindukulasuriya and Lim [9] the liquid contaminant will act as a heat sink by absorbing the thermal energy that passes from the seal jaws through both plies of film. The thermal diffusivity of vegetable oil, 0.09 x 10^{-4} m^2/s at 20^{o}C, is lower than water 1.4 x 10^{-4} m^2/s at 20^{o}C [9]. However, this study uses a salt water solution instead of water. Therefore, the heat sink effect will be greater with salt water due to its ability to absorb more thermal energy than vegetable oil. Less thermal energy passing through the liquid contaminant may affect the interface temperature of the film. The thermal diffusivity of the liquid contaminants may affect the seal strength and Hermeticity compared to the control, which has no contamination in this study.

Different oil-based and salt-based liquid contaminants have different surface tension with the film which refers to the contact area between the contaminant and film. The contact of the area of the liquid contaminant is due to the surface tension between the liquid and film. Young determined the equation for the relationship between liquid, solid, and vapor between a liquid droplet and a solid surface:

$$Y_{SV}{}^{o} - Y_{SL} = Y_{LV}{}^{o} cos\theta$$

Where, $Y_{SV}{}^{o}$ is the surface tension of the solid and vapor boundary. Y_{SL} is the surface tension of the solid and liquid boundary. $Y_{LV}{}^{o}$ is the surface tension of the liquid and vapor boundary [9,10].

When in contact with a solid surface, the contact angle θ for water is 89.51^{o} ± 1.17^{o} and vegetable oil is 29.96^{o} ± 1.2^{o} [9]. Overall, the contact angle indicates the amount of contamination that comes in direct contact with the film over an area of the film. However, the movement of the seal jaws will cause the contaminant to displace over the area of the seal. Furthermore, the surface tension and contact angle influence the displacement of the

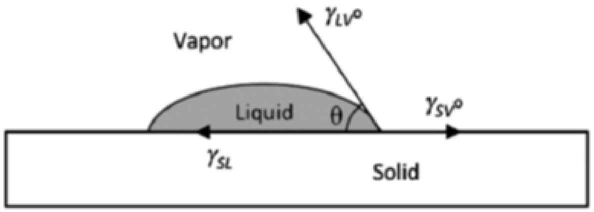

Figure 3: Liquid Contact Angle with Solid Surface

contaminant that occurs during the sealing process. Both contaminants apply the same volume of contamination, but the contaminant to film contact area will be different.

In addition to contact angle, the liquid's density is an important property for determining liquid displacement during the heat sealing process. The salt percentage used in the salt water solution is 8.2% to simulate contaminant performance more similar to salty foods. The density varies slightly for different vegetable oils, but soybean oil has a density of 0.9185 g/cm^3 at 20^{o}C [11]. According to Rodenbush et.al [12], density for vegetable oil decreases by 0.00064 g/cm^3 for every 1^{o}C increase in temperature as shown by the equation below:

$$\rho = a + b \cdot T$$

Where, ρ expresses the density in grams per cubic centimeter, a is the intercept, b is the negative slope referring to the relationship between density and temperature, and T is the temperature in Celsius. Water has a density of 0.988 g/cm^3 at 21^{o}C [13], which is greater than vegetable oil and will displace more compared to vegetable oil.

Different densities mean the contaminants will displace differently during the sealing process. Furthermore, the density indicates salt solution will displace more when the seal is made compared to vegetable oil because it has a greater density. The greater contact angle of the salt solution also shows there is less initial contact with the film for salt water for the same volume of contamination. Once the two seal jaws bring the two film surfaces together, then the water contaminant should be expected to spread over a greater area. In addition, the salt solution is expected to come in less contact with the film due to the surface tension.

1.5 Previous Testing Methods for Leaks with Seal Contamination

To test seal integrity, there is either destructive or non-destructive methods. Some destructive methods include tensile testing for seal strength, water vacuum chamber used for hermeticity testing, and dye penetration to show leaks in seal. Dye penetration is a visual inspection to check for leaks shown by a path through the seal from the inside to the outside of the bag. Matthew et. al [6] determined dye penetration is a poor method to test the presence of seal leaks because only samples exposed to excessive sealing conditions pass the test.

Non-destructive tests include ultrasonic pulse-echo or ultrasonic transmission testing for defects in the seal such as contamination. Transmission uses transmitting and receiving transducers on opposite sides of the seal. A contaminant in the seal will decrease the amplitude of the ultrasonic beam passing through the seal [14]. On the other hand, pulse-echo used a reflective pulse to test for, cracks, folds, voids, shrinks, porosity and flaking in metals [14]. Prior to Ozguler's [14] study on ultrasonic pulse-echo technique for flexible packaging, it was assumed that the technology was insensitive to test seal integrity for flexible films [14]. Ultrasonic pulse-echo used backscattered amplitude integral (BAI), which is an acoustic technology compared to optical to record the reflective sound waves to detect seal defects. Furthermore, BAI measures sound waves at 17.3 MHz and can detect any defects whether it is water or an air bubbles as long as the test is done within 10 µm range of the film [14].

1.6 Sealants and Film Characteristics

The sealant layer is the inner most layer of the packaging film that comes in direct contact with the opposing sealant layer during the sealing process. A high quality sealant has a broad sealing window and high hot tack strength [15]. A wide range of sealing temperature also allows for lower sealing temperatures without compromising the integrity of the seal. In addition, the hot tack strength refers to the film's ability to refrain from strains during its molten state [16]. A sealant with a low seal initiation temperature allows for lower process sealing temperatures, and a lower sealing temperature will use less energy than a higher sealing temperature.

Furthermore, the study uses a LLDPE commercial grade for its higher tensile strength, puncture resistance, and elongation compared to LDPE [16]. There are three polymerization processes – 1) high pressure 2) gas phase 3) slurry pressure 4) solution. More importantly, the linear low-density polyethylene sealant uses The Dow Chemical Company's constrained geometry catalyst systems (CGCT) or INSITE™ technology. INSITE™ uses Metallocene catalysts for a solution process for improved physical properties and process capabilities [17]. The improved long chain branching (LCB) of the polymer produced by INSITE technology is not found in other Metallocene technology processes. The polymers produced with LCB have an improved melt fracture resistance and uniform shear resistance process capability [17]. Lastly, the Metallocene copolymer has a lower melting point due to the increase in long chain branching to short chain branching ratio. The reduction in the comonomer short chains allows for a low seal initiation temperature.

Package performance depends on the film structure chosen for a product. In addition to providing high quality seals, the film must support the product and its expected shelf life from the time the product is packaged, followed by transportation, and lastly consumed by the consumer. A Failed hermetic seal can shorten the shelf life of a product. Even though this study observes seal strength and Hermeticity due to contamination, different food products react with the film over time. Depending on the food product application, oxygen and water vapor barriers are important characteristics of a film to ensure the shelf life of the product.

1.7 Food Product Applications

The horizontal flow wrap machine used in this study is commonly used for snack foods such as bar type foods, sliced and block cheese, cookies and other baked goods. Packaging processes are best suited for each product application based on the product's needs. For example, a vertical form, fill, and seal machine is used to pack flexible pouches with product using gravity such as shredded cheeses or bagged lettuce. On the other hand, candy bars and cookies that require more delicate handling or thermoformed trays will use a horizontal flow wrap machine. In addition, some food product applications require modified atmosphere packaging or vacuum packaging to delay the oxidation or aging process of the product. For example, vacuum packaging is commonly used for cheese packaging to eliminate the oxygen in the headspace to prevent aerobic bacteria, yeasts, and molds [18]. Trapped air in the package can quickly shorten the shelf life of the cheese, but a poor seal can also lead to oxygen passing through to the product due to leaks in the seal.

The study relates most to snack bars such as cereal bars, protein bars, and candy bars, but it can be used to relate to the greater food industry. The majority of these foods are stored by the retailer and consumer at ambient conditions, which is considered to be $23^{0}C \pm 1^{0}C$ [19].

1.8 Review Summary

Overall, the flexible packaging market is a growing and successful market especially for the food industry. Developments in film and polymerization process technologies have made film packaging more desirable and applicable to many food industries. Previous work studied the seal strength of flexible pouches with seal contamination, but this study further determines the effect on seal through liquid contamination and temperature over time. Sealing temperature and dwell time are the two primary sealing factors to produce a quality seal. As mentioned previously, seal jaw pressure has little effect on the quality of the seal. Lastly, sealing through liquid contamination may be detected with current quality control processes, but this study investigates the impact on the seal's properties.

The objective of the study is determining the effect of liquid contamination found in the seal area on Hermeticity and seal strength for flexible pouches with linear low-density polyethylene sealant. First, the study must determine if liquid contaminants perform differently at different sealing temperatures and dwell times. In addition, the study determines if time and storage temperature affect the performance of liquid contamination in the seal area. The optimal sealing condition with the highest Hermeticity pass rate is determined based on statistical analysis. Furthermore, a shelf life study is used to ensure Hermeticity does not change over time. If there is a seal leak two days after the package is produced, then it should also show a seal leak fourteen days after production. It was also important to observe the difference in results for both Hermeticity and seal strength. The three test categories are salt water for sodium based foods, vegetable oil for oil-based foods, and non-contaminated seals (control).

2.0 MATERIALS AND METHODS

2.1 Film Structure

The film used in this study was provided by The Dow Chemical Company and it is a commercial grade film currently used by the snack food industry. The film is a DOWLEX™ 2045G LLDPE 40.64 µm film. The PET film is a laminate that is commonly used in films for improved puncture resistance, and barrier properties. An adhesive was used to adhere the PET laminate to the film. Overall, the film is tested for performance in addition to the sealant performance since it comes in direct contact with the contaminant.

The linear low-density polyethylene (LLDPE) sealant, with the trademark name ELITE™ 5400G, has a 0.916 g/cm^3 density and has a low seal initiation temperature, 90^{o}C. The sealant's puncture resistance equals 107 N and 5.5 J. Three film rolls were supplied for the study from the beginning of Phase 1 to Phase 2.

Figure 4: Film Structure

2.2 Equipment

The experiment uses the horizontal form fill and seal (HFFS) machine manufactured by Delfin (Figure 5), and a Dow specialty film with a chosen sealant grade commonly used for commercial snack food applications. For Hermeticity testing, the Test-A Pak integrity tester is a large cylindrical water tank with a lid that submerges one inch under water. The Test-A Pak is a vacuum chamber to inflate the sample bags and visually observe for seal leaks.

The Testometric tensile tester is a pneumatic system for seal strength testing. A Raytek temperature gun is used to test the actual temperature of the environmental chambers and the actual temperatures of the sample bags. The two environmental chambers used for conditioning temperatures are manufactured by Darwin Chamber Company. Lastly, a JDC precision sample cutter, manufactured by Thwing-Albert Instrument Company is used for cutting the specimen samples for seal strength testing from the sample bags.

Figure 5: Overall System

2.3 Consistency

The system must allow for consistent contamination from top to bottom seal for all types of contamination. The stream of contamination must be consistent from sample bag to sample bag to ensure data results are accurate. To measure consistent contamination, the width or thickness of the contamination was measured during preliminary testing. The width measurements of the stream was recorded for the front and back seals and the midpoint along the contamination stream. However, there was some tolerable variation allowed from sample bag to sample bag since the measurements were made visually by human eye. The acceptable coefficient of variation was considered to be less than 15%. A preliminary trial was conducted to measure the contamination and to test the contamination system. The flow rate of contamination may vary between contamination types, so the flow rate was measured for each contamination as well as the width or thickness of the contamination stream.

2.4 Contamination System

The contamination stream falls along the path of the fin seal to contaminate the T-point, which is the most critical point of failure on the package. The T-point is chosen for point of contamination since the seal jaws will be sealing through four layers of film. The decision to contaminant on top of the fin seal was chosen because it is most difficult to seal through four layers of film at the optimal condition for temperature and dwell time.

The three test categories for this study are vegetable oil and a salt water solution, and non-contamination which is the control. Vegetable oil was used to simulate for oil based products that will be in a flexible film packaged on a horizontal flow wrap machine. Salt Water was used to simulate snack food products that are sodium based packaged in a film on a horizontal flow wrap machine. *Pure Wesson 100% Natural Soybean Oil* brand was used in the study for the vegetable oil contaminant. No additives such as water were added to the vegetable oil in the study. *Morton Salt* brand was mixed with water for the sodium water contaminant. The salt percentage in water was 8.2% or 41.92 grams per 465.16 ml of water. The percentage of sodium water was chosen based on preliminary work to maximize the amount of salt in the solution with semi-dissolved characteristics. Once the solution was mixed with a tongue depressor for two minutes, the sodium was dissolved into the water but the salt grains remained visible in the contamination stream.

A system was created on the machine that uses a mechanical syringe pump, 60 ml syringe, 1/4" OD

0.170” ID clear vinyl tubing to attach the syringe to the 3/16” OD 0.148” ID steel tube (three feet), one brass adapters (male), and one brass swivel straight nozzles (female) (Figures 6 & 7). The system forces one stream of contamination inside the bag by exposing the nozzle right after the fin seal is created and before the two end seals are made. This allows for contamination to be contained in the bag during the tube form as well as contaminate the end seals.

The syringe pump has a vinyl flexible tube (o.d.) which is extended to a stainless steel tube on the opposite end, which has a single nozzle to release the contaminant onto the film (Figure 7). The fin seal was created through rollers underneath the conveyor belt, which creates the tube of film. The nozzle hovers over the fin seal once it is created and passes the heated rollers underneath the conveyor belt. The nozzle hovers to avoid preventing the film from moving forward, but the tip of the nozzle still comes in direct contact with the film (Figure 7).

The system has two tongue depressors attached to the right side of the stainless steel tube once the fin seal starts to form to prevent the tube of film to

Figure 6: Mechanical Syringe Pump Setup

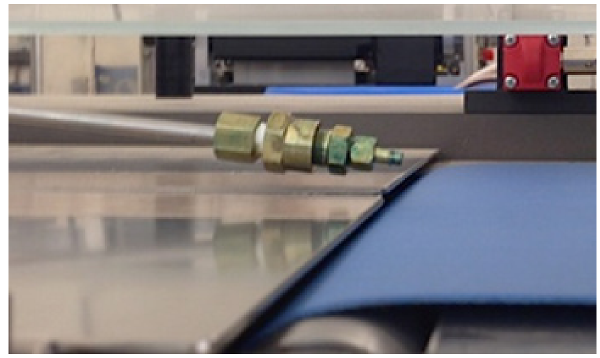

Figure 7: Contamination Release Point

shift to one side of the conveyor belt. It is observed in the preliminary study that the tube will shift to the left causing a corner leak on the same side for the front and end seal. After the addition of the tongue depressors, the detection of corner leaks during the Hermeticity test was reduced.

The stainless steel tube is placed through a wooden block that is attached to the inner former to keep it from moving freely during production. The wooden block is held in place to avoid unwanted movement during production of the sample bags.

2.5 Method

2.5.1 Phase 1 and Phase 2 Description

Phase 1 has 6 test categories that were stored at ambient temperature (20^{o}C) for two hours to ensure that the polymer chains have achieved chemical stabilization [5] before they went through hermeticity testing. Phase 2 uses the same test categories as Phase 1, but it introduces storage conditions over a 10 day period.

2.5.2 Phase 1

There were a total of nine conditions (temperature and dwell time combinations) (Table 1) that were used in Phase 1 which included different temperatures and dwell times. Phase 1 conducts Hermeticity testing at all nine conditions to determine the most optimal sealing condition.

Phase 1 included two contaminants – vegetable oil and salt water at all the above conditions (Table 1). Non-contaminated bags will be the control in the study. A total of 13 samples for each bag type were made at each condition to account for discarded samples during Hermeticity testing. The discarded samples can result from poor fin seals, unwanted crease or folds in the seal, and sample bags bursting open under vacuum pressure.

Table 1: Sealing Condition Combinations

Temperature		Dwell Time (sec)		
		D1	D2	D3
T1	120ºC	0.2	0.3	0.4
T2	140ºC	0.2	0.3	0.4
T3	160ºC	0.2	0.3	0.4

Phase 1 was conducted over a six day period. One contaminant type was randomly selected for each day over the six day period. Therefore, the nine machine conditions for each type of contaminant are randomly divided into two days. The random order is shown in Table 2.

2.5.3 Hermeticity Testing

Hermeticity of the Flexible Bags were tested in accordance to ASTM Standard D3078 – T*est Method for Determination of Leaks in Flexible Packaging by Bubble Emission* [20]. Each sample bag was placed in the vacuum chamber with an attached cover plate immersed under water by one inch, with the fin seal faced down.

Table 2: Test Schedule for Phase 1

Categories		Condition				
		1	2	3	4	5
Day 1	Control	T1, D2	T2, D3	T3, D1	T2, D2	T3, D3
Day 2	Salt Water	T2, D3	T1, D3	T2, D1	T3, D3	-
Day 3	Vegetable Oil	T1, D3	T3, D1	T2, D1	T2, D3	-
Day 4	Salt Water	T1, D2	T1, D1	T3, D1	T2, D2	T3, D2
Day 5	Control	T2, D1	T3, D2	T1, D3	T1, D1	-
Day 6	Vegetable Oil	T3, D2	T1, D1	T3, D3	T2, D2	T1, D2

Air bubbles can be of different sizes depending on the total area of the leaked seal. According to ASTM Standard D3078, a small bubble will release ½ ml of air over 365 seconds [20].

As seen in Table 3 that small leaks will produce 0.41 ml per 30 seconds; medium leaks will produce will release 0.1826 ml per 30 seconds; and large leaks will release 0.574 ml per 30 seconds. In addition, the bubbles must continuously surface from one seal point to be considered a failed hermetic seal. Using Table 5 from ASTM D3078 [20], three continuous bubbles released over 30 seconds was considered a failed hermetic seal. There are different size leaks which will release different sizes. However, in this study the size of the bubbles cannot be determined without access to Helium leak detector, which is explained in ASTM D3078 [20].

Table 3: Bubble Size Categories for Hermeticity [20]

Size (He)	Average	Sr	SR	r	R
Big 6E-02	26.11	2.667	3.918	7.467	6.122
Medium 7E-03	82.11	4.073	6.196	11.406	13.019
Small 3E-03	365	18.963	32.549	53.096	69.962
Very Small 1E-04	0.037	0.192	0.192	0.00	1.235

Hermeticity of the seals was tested by using a vacuum chamber that places the bags into a contained tub of water and the pressure was brought down to 22.0 in Hg. The standard ASTM D3078 suggests three vacuum levels – low vacuum (12.5 ± 0.5 in. Hg), medium vacuum (18.5 ± 0.5 in. Hg), and high vacuum (24.5 ± 0.5 in. Hg). The preliminary work tested the three vacuum levels with the film used in the experiment as well as different films with different film structures. The different films required different vacuum test levels, and it was determined that the suggested vacuum levels were inadequate for the test. For the film used in the experiment, the high vacuum level caused every test bag to burst open in the vacuum chamber and the medium vacuum level did not apply enough vacuum pressure to inflate the bags. Therefore, the preliminary test included testing vacuum pressures between 24.5 in. Hg and 18.5 in. Hg. As a result, 22.0 in. Hg seemed to be the best fit for this type of film and bag structure. The size of the test bags and the film structure seem to be two factors that influence the vacuum pressure for Hermeticity testing.

Overall, the vacuum pressure causes the bag to expand, and allows bubbles to form at the leaking points at the seal. The bubbles indicate a failed hermetic seal and no bubbles indicate a pass hermetic seal. The bags were placed in the hermetic fish tank for 30 seconds, which is a sufficient time period to observe bubble formations. Bubbles may tend to form that are trapped at the surface and along the surface of the seal. Furthermore, a failed hermetic seal will show continuous bubbles from a leaking point.

Bubble formation may occur at different points on the seals of the sample bags. The locations of the leaks were recorded and categorized based on type of leak. Bubbles forming at the corners of the seal will be considered a "pass" in this study since the corners are not subject to contamination (Table 4). However, corner leaks that also have a leak at the T-point point will be considered a "fail" (Table 4).

Figure 8 shows the critical point where the seal jaws must seal through four layers of film. For a successful seal to be produced at the T-point, the polymer chains in the sealant layer should reach their molten stage and completely entangle. However, a low sealing temperature and dwell time can limit the chains from entangling and result in a failed hermetic seal.

Table 4: Hermeticity results based on leak location

Leak Location	Hermeticity Result
No leaks	True Pass
At corners	Pass
At T-point	True Fail
At T-point and corners	True Fail
At contamination area (near T-point point)	True Fail

2.5.4 Phase 2

Phase 2 tested the effect of storage temperature, time, and contaminants with the film using the optimal packaging sealing condition determined in Phase 1. Control samples were made for each contaminant's packaging sealing condition if the optimal packaging sealing condition is different for each contaminant. However, Phase 1 results indicate that both contaminants have an optimal

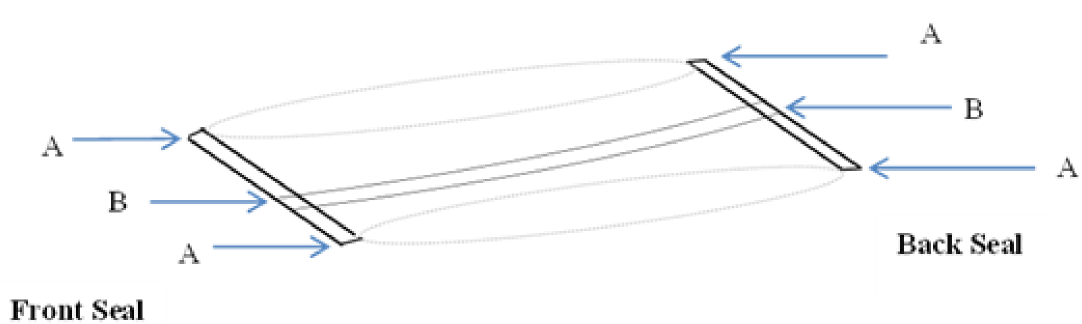

Figure 8: Critical Points for Bag Samples
A = Seal Corners; B = T-point Point

packaging condition of 140ºC sealing temperature with 0.3 seconds dwell time. Table 5 shows a randomized production schedule of samples for Phase 2 using Microsoft Excel.

The sample bags were produced over a three day period in a complete random block design schedule. Once all bags were produced each day, they were randomly placed in the environmental chambers and chosen at random each day for sample testing. In addition, the bags were randomly placed during production into each of the eight corrugated boxes.

Ten samples for Hermeticity and five samples for seal strength were taken each day on Day 2, 6, 10 and 14 from each of the refrigerated and ambient temperature chambers (Table 6). A 14 day period was chosen to assume the average time period between packing and production of a product until it reaches a consumer.

Each contaminant had 120 samples randomly placed in 12 boxes with an additional 12 to 24 bag samples to account for necessary discarded samples throughout Phase 2. In addition, the 12 boxes were randomly chosen for each conditioning temperature using Microsoft Excel. Lastly, each of the 6 boxes were randomly chosen for Hermeticity and seal strength, and randomly placed in each chamber.

The temperature and relative humidity of each chamber was recorded on each data collection day at the beginning of testing. The actual chamber temperature was measured using a Raytek temperature hand gun, but no accurate tool was used to measure actual relative humidity. For each testing period, one box for Hermeticity and one box for seal strength was removed from the chamber and opened immediately to determine the actual temperature of the sample bags. However, each testing

Table 5: Phase 2 Production Schedule

Date	Contaminants	Total Sample Bags Produced	(+10% more samples)	Total
19/2/2014	Salt Water	120	12	132
20/2/2014	Vegetable Oil	120	12	132
21/2/2014	Control	120	12	132

Table 6: Phase 2 Sample Size for Each Collection Day

	Hermeticity Testing Sample Size		Seal Strength Testing Sample Size		
Testing Procedure	Refrigerated	Ambient	Refrigerated	Ambient	Total
Day 2	10	10	5	5	30
Day 6	10	10	5	5	30
Day 10	10	10	5	5	30
Day 14	10	10	5	5	30
Total	40	40	20	20	

period required only five bags for seal strength testing, so the seal strength box was returned to its placement in the designated chamber after the necessary sample bags were removed from the box for testing. The boxes chosen for Hermeticity and seal strength for each test day were randomly selected.

2.5.5 Seal Strength Testing

In addition to Hermeticity, the seal strength was tested to determine if there is a significant difference between contaminated and non-contaminated seals. The purpose of testing the seal strength was to determine the consistency of seal strength from sample bag to sample bag with contamination and without contamination. It was also used to validate the sealing conditions for packaging production. According to ASTM F88 – Standard Test Method for Seal Strength of Flexible Barrier Materials, the sample for peel force will be one inch wide and three inches long from the end point of the seal [3]. The standard does not indicate a necessary sample number, so ten samples were chosen for each condition.

The unsupported seal strength test was used for this study (Figure 9). It is not expected to have another force affecting the seal strength as is shown in the above Figure 9. Each leg or unsealed section is fastened to the top and bottom grip on the tensile tester. The seal is tested at a rate of 30.48 cm/min. and the maximum force to failure was recorded. The average seal strength (n) is the average force per unit width of seal at failure. [3,5,6]

2.5.6 Determining Optimal Packaging Conditions

The Hermeticity test was conducted to show the pass percentage of each contaminant at the different conditions. It was assumed that there will be a difference between contaminated and non-contaminated seals. In addition, the contaminated seals have a lower hermetic pass rate than non-contaminated bags. The optimal packaging condition considers

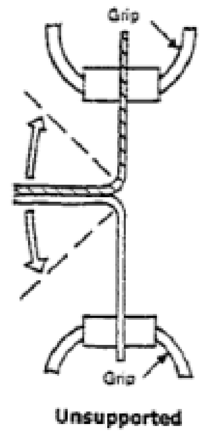

Figure 9: Specimen Setup for Unsupported Seal Strength Test

the temperature and dwell time of the end seals that gives the highest pass rate that remains insignificantly different than non-contaminated bags. Lower the seal temperature, lower the amount of energy needed to package the bags, and lower dwell time indicates a faster production speed. In addition to determining the statistical difference within one condition, it is also important to determine if there is a statistical difference between the contaminated hermetic pass percentage with a lower dwell time to a higher dwell time. If between the two dwell times at the optimal seal temperature is insignificant, then the lower dwell time would be used. However, if the hermetic pass percentage is significant between the two dwell times, then the longer dwell time will be the optimal condition. One sealing condition of one temperature and dwell time was used for each contaminant type for Phase 2.

After determining the optimal packaging sealing condition for each contaminant, Phase 2 tested the performance of the contaminated versus non-contaminated seals over a shelf life of 14 days. A two week period was chosen because this is the common time period a package is on the shelf for these product types. The same size bags were made using the HFFS machine, but only using the optimal packaging condition for each contaminant. Non-contaminated bags were made as the control for each of the contaminant's optimal seal condition. The bags were prepared at ambient temperature of approximately 22ºC and 33% RH. Further, each contaminant was tested for two types of environmental conditions – refrigerating condition at 5ºC and 85% humidity and ambient conditions of 23ºC and 50% humidity [19].

The second phase of the study will test each contaminant's performance at standard conditions at 23ºC ± 1ºC (73.4ºF ± 2ºF) and 50% ± 2% relative humidity, and refrigerated conditions at 5ºC ± 2ºC (41ºF ± 4ºF) and 85% ± 5% relative humidity [19]. The conditioning temperatures are set at a constant for a two week test period to test the effect of temperature on hermeticity and seal strength for the two contaminant types.

3.0 RESULTS AND DISCUSSION

3.1 Contamination consistency results from Phase 1 and 2

Throughout preliminary work , Phase 1 and Phase 2 of the study, the consistency of contamination from sample bag to sample bag was measured. A ruler was used to measure the width of the contamination stream near the front and back seals as well as the midpoint of the stream. Overall, vegetable oil had an average coefficient of variation of 0.217 which is greater than 0.15. As mentioned previously, 0.15 coefficient of variation was the goal of the study to have almost no variation from sample bag to sample bag. On the other hand, the salt water contaminant had a higher coefficient of variation of 0.211, which is greater than the desired 0.15 coefficient of variation. The greater C.O.V of salt water could be due to the density and contact angle of the solution. In comparison to vegetable oil, the salt water solution is more prone to move during production. A greater amount of sample bags were measured for salt water to measure its incline to move away from the fin seal path. Even though the salt water solution has a higher coefficient of variation, both contaminants were considered to have an acceptable consistent stream.

3.2 Phase 1 Results

The results showed that an increase in temperature increased the pass rate within 120ºC to 160ºC (Figures 10, 11 & 12). For each temperature, there was also an increase in pass rate as the dwell time increases (Figures 10, 11 & 12). The Hermeticity test observed the leaks in the seals for the sealing conditions and what type of leaks were occurring (Table 7,8 & 9). Contaminants fell only along the fin seal and contaminated the T-point, so any fails that are not along the T-point were considered a pass such as corner leaks. It was found that some leaks occurred at the corners of the seal which were considered pass but not a true pass since they were not contaminated. T-point failures were what were compared in this study, so all corner leaks were considered a pass.

3.2.1 Hermeticity Results by Sealing Condition

3.2.1.1 Results at 120ºC Sealing Temperature

From the results of 120ºC sealing temperature (Figure 10 and Table 7), it can be inferred that the results for 0.3 seconds dwell time are inconclusive since the pass rate for both vegetable oil and salt water is greater than the control. Therefore, 120ºC with 0.3 seconds dwell time was considered an optimal packaging condition for Phase 2. The error for 0.3 seconds dwell time could be a result of the small sample size of 10 replicates. It is possible that a larger sample size may eliminate the error. However, the pass rate for 120ºC and 0.3 seconds dwell time has a low pass rate for all contaminants and control compared to the other conditions in Phase 1 (Figure 10). The results indicate that there is a large difference between the contaminants and the control for 0.2 seconds and less of a difference for 0.4 seconds dwell time (Figure 10). Overall, there is an increase in pass rate as the dwell time increases for 120°C sealing temperature. As mentioned previously, the pass rate also includes leaks only at the corners during the test.

3.2.1.2 Results at 140°C Sealing Temperature

From the results shown in Figures 11 and Table 8 for 140°C sealing temperature, it can be seen there is no test category that had a consistently higher pass rate than the other test categories or a consistently lower pass rate at all dwell times. The condition for the highest pass rate that is statistically significant for all test categories is 140°C and 0.3 seconds dwell time. Also, 140°C sealing temperature has a higher pass rate than 120C for all dwell times (Figures 10 & 11).

3.2.1.3 Results at 160°C Sealing Temperature

At 160°C sealing temperature and 0.2 seconds dwell time, there is a difference between the

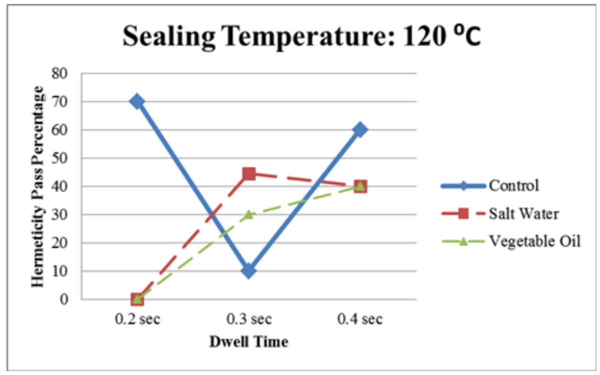

Figure 10: Contaminant Pass Rates by Dwell Time for 120°C Sealing Temperature

Table 7: 120°C Sealing Temperature at various Dwell Times

Seal Temperature 120°C	Dwell Time (Secs)								
	0.2			0.3			0.4		
	Pass	Fail	Fail Type	Pass	Fail	Fail Type	Pass	Fail	Fail Type
	7	-	corners	1	3	T-point & corners	7	-	T-point & corners
Control	-	3	T-point	-	-	T-point	-	3	T-point & corners
	-	-	-	-	6		-	-	
Total	7	3		1	9		7	3	
	0	10	T-point & corners	2	1	T-point	3	3	T-point
Salt Water	-	-	-	2	-	corner	1	-	corner
	-	-	-	-	4	T-point & corners	-	3	T-point & corners
Total	0	10		4	5		4	6	
Vegetable Oil	0	10	T-point & corners	1	7	T-point	4	6	T-point
	-	-	-	2	-	corner	-	-	-
Total	0	10		3	7		4	6	-

contaminants and the control (Figure 12 and Table 9). The control has a 40% higher pass rate than salt water and 30% higher pass rate than vegetable oil. Vegetable oil had one failed sample that showed a leak at the contaminate area near the T-point (Table 9). Although, the contaminant lies along the T-point, the contaminant spread within the area near the T-point when the two surfaces came together. Even though this total area is not measured in this study, the initial thickness of the contamination was recorded.

For seal condition 160ºC and 0.3 sec dwell time, the salt water contaminant had one failed sample that showed leaks from the contaminated area near the T-point (Table 9). The control had nine samples that were true passes, and one sample that showed a leak at the corners. Salt water and vegetable oil contaminants each had four samples that failed at the T-point. In comparison to the previous condition of 160ºC and 0.2 seconds dwell time, pass rates increased for all test categories.

At 160ºC sealing temperature and 0.4 seconds

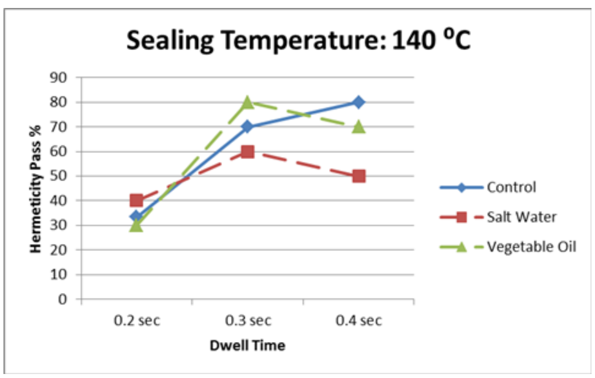

Figure 11: Contaminant Pass Rates by the Dwell Time for 140°C Sealing Temperature

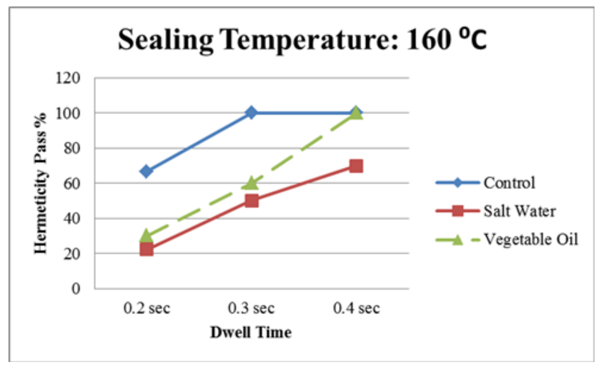

Figure 12: Contaminant Pass Rates by the Dwell Time for 160°C Sealing Temperature

Table 8: 140°C Sealing Temperature at various Dwell Times

Seal Temperature 140°C	Dwell Time (Secs)								
	0.2			0.3			0.4		
	Pass	Fail	Fail Type	Pass	Fail	Fail Type	Pass	Fail	Fail Type
Control	2	3	T-point	6	2	T-point	7	2	T-point
	-	3	T-point & corners	-	1	T-point & corners	1	-	Corner
	1	-	Corner	1	-	Corner	-	-	
Total	3	6		7	3		8	2	
Salt Water	3	6	T-point	6	4	T-point	5	5	T-point
	1	-	Corner	-	-	-	-	-	-
Total	4	6	-	6	4		5	5	
Vegetable Oil	3	7	T-point	6	2	T-point	7	3	T-point
	-	-	-	2	-	Corner	-	-	-
Total	3	7		8	2		7	3	

dwell time, vegetable oil contaminant and the control had all ten samples pass with no leaks (Table 9). The salt water contaminant had the lowest pass rate of 70% with two samples failed at the T-point point and one sample failed with T-point and corner leaks.

3.2.2 Phase 1 Statistical Analysis

A binary logistic regression was used in this study to analyze the odds ratio for the Hermeticity pass rate. The results show the odds of passing between the different sealing temperatures and the dwell times. The alpha (α) equals 0.05, which is the probability of rejecting the null hypothesis.

Phase 1 Null and Alternative Hypotheses:

HO(temp) :There is no difference in Hermeticity pass rate between 120°C, 140°C, and 160°C.

HA(temp) :There is a difference in Hermeticity pass rate between 120°C, 140°C, and 160°C

HO(dwell time) :There is no difference in Hermeticity pass rate between 0.2s, 0.3s, and 0.4s

HA(dwell time) :There is a difference in Hermeticity pass rate between 0.2s, 0.3s, and 0.4s

HO(contaminant) :There is no difference in Hermeticity pass rate between the control and salt water and vegetable contaminants.

HA(contaminant) :There is a difference in Hermeticity pass rate between the control and salt water and vegetable oil.

Table 10 compares 120ºC and 160ºC to 140ºC sealing temperature. The table shows that 160ºC sealing temperature has greater odds of passing the Hermeticity test compared to 140ºC. For example, for every 10 sample bags that have a hermetic seal with 140ºC sealing temperature 16 sample bags will have a hermetic seal when accounting for dwell time and contaminants. However, the p-value of 160ºC equals 0.166. Therefore, we are 95% confident that there is not enough evidence to conclude that there is a difference in pass rate between 160°C and 140°C when accounting for the effect of dwell time and contamination. In addition, 120°C sealing temperature has a lower pass rate than 140°C because the odds ratio, 0.32 is less than one. The odds ratio

Table 9: 160°C Sealing Temperature at various Dwell Times

Seal Temperature 160°C	Dwell Time (Secs)								
	0.2			0.3			0.4		
	Pass	Fail	Fail Type	Pass	Fail	Fail Type	Pass	Fail	Fail Type
Control	5	3	T-point	9	0	-	10	0	-
	1	-	Corner	1	-	Corner		0	-
Total	6	3		10	0		10	-	
Salt Water	2	5	T-point	5	4	T-point	7	2	T-point
	-	2	T-point & corners	-	1	Contamination		1	T-point & corners
Total	2	7		5	5		7	3	
Vegetable Oil	3	5	T-point	6	4	T-point	10	0	-
	-	1	T-point & corners	-	-	-	-	-	-
		1	Contamination	-	-		-	-	-
Total	3	7		3	7		10	0	

indicates that 120°C sealing temperature has the odds of producing a hermetic seal 0.32 compared to every control sample has a hermetic seal. The p-value for 120°C compared to 140°C equals 0.001. Therefore, we are 95% confident that there is a difference in pass rate between 120°C and 140°C when accounting for the effect of dwell time and contamination. Vegetable oil and salt water have a lower pass rate than the control, but vegetable oil has greater odds of passing than salt water. The odds for vegetable oil and salt water are 0.45 and 0.30, which are both less than one. If the odds ratio was greater than one, then the contaminants would have greater odds for a hermetic seal than the control. The p-values for both salt water and vegetable oil are 0.001 and 0.021, so there is enough evidence to reject the null hypothesis, HO(contaminant). Therefore, we are 95% confident that there is enough evidence to conclude that the contaminants will have a lower pass rate compared to the control when accounting for the effect of dwell time and sealing temperature.

The dwell time of 0.2 seconds compared to 0.3 seconds has an odds ratio less than one and a p-value less than 0.05 (Table 10). Therefore, we are 95% confident that there is a difference between 0.2 seconds and 0.3 seconds when accounting for contamination and sealing temperature. Also, the dwell time of 0.4 seconds compared to 0.3 seconds has a p-value that is 0.054 which is slightly more than 0.05 (Table 10). Since it is more beneficial to use a shorter dwell time for production, 0.4 will not be used for the dwell time in Phase 2. Therefore, we are 95% confident that there is not a significant difference between 0.4 seconds and 0.3 seconds dwell time.

Table 11 compares 140°C and 160°C to 120°C, and 0.3s and 0.4s to 0.2s. In comparison to 120°C, both 140°C and 160°C have an odds ratio that is greater than one. The p-value for both temperatures

Table 10: Phase 1 Binary Logistic Regression Analysis

TEMP (Ref. 140°C)	Odds Ratio*	95% C.I.	P-Value
120°C	0.32	(0.16, 0.62)	0.001
160°C	1.6	(0.82, 3.10)	0.166
DWELL TIME (Ref. 0.3 s)			
0.2 seconds	0.33	(0.17, 0.65)	0.001
0.4 seconds	1.92	(0.99, 3.71)	0.054
Contaminant (Ref. Control)			
Salt Water	0.3	(0.15, 0.60)	0.001
Vegetable Oil	0.45	(0.23, 0.89)	0.021

**The odds ratio refers to the category associated with the odds ratio compared to the reference category*

are less than 0.05 and can reject the null hypothesis, HO(temp). Therefore, we are 95% confident that there is a difference between 120°C and 140°C as well as 120°C and 160°C when accounting for the effect of dwell time and contamination. Looking at the dwell time comparison, 0.3s dwell time and 0.4s dwell time have an odds ratio of 3.01 and 5.76. The values are greater than one, which imply that 0.3s and 0.4s dwell time have greater odds for passing Hermeticity than 0.2s dwell time. Furthermore, the p-values for both dwell times are less than 0.05 and can reject the null hypothesis, HO(dwell time). Therefore, we are 95% confident that there is a difference between 0.2 seconds and 0.3 seconds as well as 0.2 seconds and 0.4 seconds for dwell time when accounting for the effect of sealing temperature and contamination. The contaminants' odds ratio indicates vegetable oil will have a higher pass rate compared to salt water when comparing against the performance of the control. The p-values for salt water and vegetable oil are 0.001 and 0.021, and can reject the null hypothesis, HO(contaminant). Therefore, we are 95% confident that there is a difference in pass rate for both contaminants compared to the control when accounting for the effect of dwell time and sealing temperature.

The interaction between two factors indicates one factor is affected by the other. If there is a significant p-value for the interaction (less than 0.05), then there is an association between the two factors when determining the pass rate. For example, if the p-value for the interaction between 120°C, 0.2s and 140°C, 0.3s is less than alpha (α=0.05) then there is an association between the temperature and dwell time when testing Hermeticity.

The p-values for the interactions shown in Table 12 indicate that we are 95% confident that there is not enough evidence to conclude that there is an association between sealing temperature and contaminant type, dwell time and contaminant type, and

Table 11: Phase 1 Binary Logistic Regression Analysis

TEMP (Ref. 120°C)	Odds Ratio*	95% C.I.	P-Value
140°C	3.13	(1.61, 6.08)	0.001
160°C	5	(2.51, 9.95)	0
DWELL TIME (Ref. 0.2 s)			
0.3 seconds	3.01	(1.55, 5.84)	0.001
0.4 seconds	5.76	(2.88, 11.53)	0
Contaminant (Ref. Control)			
Salt Water	0.3	(0.15, 0.6)	0.001
Vegetable Oil	0.45	(0.23, 0.89)	0.021

**The odds ratio refers to the category associated with the odds ratio compared to the reference category*

Table 12: Binary Logistic Regression Analysis Results with Interactions

TEMP (Ref. 140°C)	Odds Ratio*	95% C.I.	P-Value
120℃	3.13	(1.61, 6.08)	0.001
160℃	5	(2.51, 9.95)	0
DWELL TIME (Ref. 0.3 s)			
0.2 seconds	0.58	(0.14, 2.45)	0.580
0.4 seconds	1.66	(0.36, 7.57)	1.66
Contaminant (Ref. Control)			
Salt Water	1.51	(0.35, 6.48)	1.51
Vegetable Oil	1.90	(0.42, 8.53)	1.90
TEMP*CONTAMINANT (Ref. 140°C)			
120*Salt Water	0.70	(0.15, 3.34)	0.700
120*Vegetable Oil	0.360	(0.07, 1.88)	0.36
160*Salt Water	0.100	(0.01, 0.67)	0.10
160*Vegetable Oil	0.18	(0.03, 1.29)	0.18
SWELL TIME*CONTAMINANT (Ref. 0.3 s)			
0.2*Salt Water	0.280	(0.05, 1.49)	0.280
0.2*Vegetable Oil	0.200	(0.04, 1.14)	0.200
0.4*Salt Water	0.240	(0.04, 1.38)	0.240
0.4*Vegetable Oil	0.630	(0.3, 3.9)	0.630
TEMP*DWELL TIME (140°C*0.3s)			
120°C*0.2 seconds	3.05	(0.58, 16.04)	3.05
120°C*0.4 seconds	3.00	(0.62, 14.43)	3.00
160°C*0.2 seconds	1.05	(0.19, 5.76)	1.05
160°C*0.4 seconds	7.69	(1.06, 55.99)	7.69

**The odds ratio refers to the category associated with the odds ratio compared to the reference category*

sealing temperature and dwell time. Moreover, the significance of the interaction does not change the optimum sealing condition for Phase 2 of the study.

3.2.3 Phase 1 Summary Findings

Vegetable oil and salt water pass rates increase from 0.2 second to 0.4 seconds dwell time. The control showed no difference between 0.3 second and 0.4 seconds since all 10 sample bags passed with no leaks. At 0.2 seconds dwell time, vegetable oil contaminant showed one sample that leaked during Hermeticity from the seal area that was contaminated near the T-point point. In addition, salt water had one sample at 0.3 seconds that failed due to leaking from the contaminated area near the T-point. Even though the study aims to observe the performance of the T-point, the stream of contamination spreads across the seal area near the T-point. Furthermore, the force from the seal jaws to bring the two film surfaces together causes the contaminant to spread in the seal area.

Overall, the binary logistic regression analysis indicates the optimal sealing condition for all contaminant types is 140°C and 0.3s dwell time. The binary logistic regression analysis in Table 12 shows there is not enough evidence to conclude there is a difference in pass rate between 140°C and 160°C sealing temperature and 0.3s and 0.4s dwell time. However, there was enough evidence to conclude that there is a difference between 120°C and 140°C sealing temperature and 0.2s and 0.3s dwell time. Furthermore, the analysis indicates 120°C sealing temperature has a lower pass rate compared to 140°C sealing temperature. In addition, the analysis shows 0.2s dwell time has a lower pass rate compared to 0.3s dwell time.

3.3 Phase 2 Results

Phase 2 tested the effect of temperature, time and contamination, the considered parameters of this Phase. Each contaminant was tested four times over the 14 day shelf life study on Days 2,6,10 and 14. On each day, it was expected to test at least 10 sample bags for Hermeticity in order to have an adequate representation for each contaminant type. Unfortunately, even with 10-20% more samples than what was needed each day, some sample sizes were less than 10 due to wrinkles in the seal and insignificant bag inflation during Hermeticity testing. To test a hermetic seal, the sample bag must fully expand in the vacuum chamber. In addition to Hermeticity testing, at least five sample bags were tested each day for each temperature for seal strength. Each sample bag had two replicates for front and back seal, which provided 10 to 12 samples for each conditioning temperature on each day. However, samples were only reported in the results if the failure mode was peel or a combination of peel and material failure.

3.3.1 Phase 2- Hermeticity Results and Statistical Analysis

Hermeticity was tested for both ambient and refrigerated condition during each testing period – Days 2,6,10, and 14 (Figures 13 &14). Hermeticity testing was conducted to determine if Hermeticity had a significant change over time due to contamination.

The binary logistic regression analysis (Table 13) indicates that salt water will likely have a lower pass rate when associated with the pass rate of the control. For example, salt water is associated with 0.64 odds of having a hermetic seal compared to the control. In addition, vegetable oil is associated with 1.09 odds of having a hermetic seal compared to the control. Therefore, vegetable oil has a similar pass rate compared to the control since the odds ratio is close to 1. However, the p-value for both salt water and vegetable oil contaminants is 0.175 and 0.792. Therefore, we are 95% confident that when accounting for the effect of storage temperature and time there is no significant difference between salt water and vegetable oil contamination compared to the

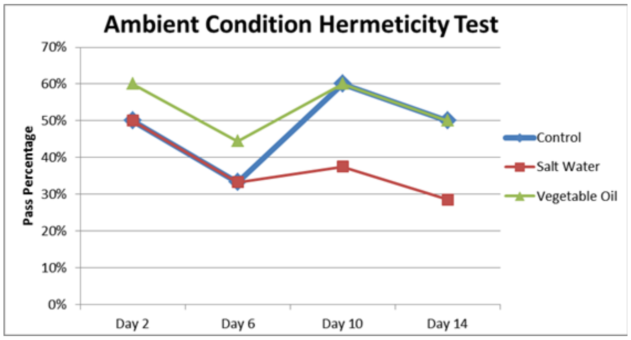

Figure 13: Phase 2 Ambient Condition Hermeticity Results for All Contaminants

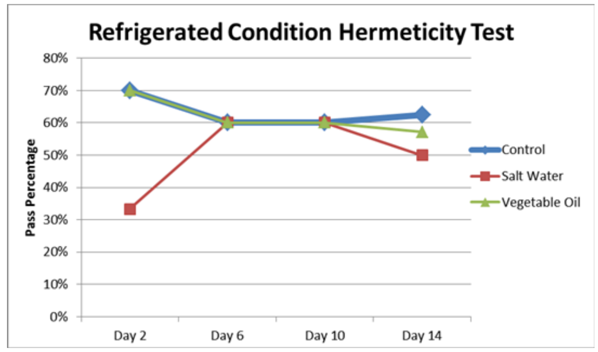

Figure 14: Phase 2 Refrigerated Condition Hermeticity Results for All Contaminants

control. Also, the refrigerated conditioning temperature is associated with greater odds of 1.63 of having a hermetic seal compared to ambient conditions.

3.3.2 Phase 2- Seal Strength Results and Statistical Analysis

Seal strength was tested for both ambient and refrigerated condition during each testing period – Days 2,6,10, and 14 (Figures 15 & 16).As mentioned in the methods subchapter, each sample bag had two replicates to represent the average performance of the sample bag using the average of the front and back T-point seals. However, if the seal strength resulted with an insignificant peel failure, then the results of that sample were not included in the represented data. Furthermore, only one seal will represent the seals strength of a sample bag if one of the seals resulted in a material failure. The results of the seal strength failure modes are divided into the categories shown in Table 14.

The Seal Strength Failure Modes for all three test categories did not show any trend in type of failures for any category. For example, no category resulted in a greater amount of peel failure compared to the other categories. In addition, no category had a shift from peel to material failure or vice versa.

Table 13: Binary Logistic Regression Analysis for Phase 2 Hermeticity Testing

Contaminant(ref. Control)	Odds Ratio	95% C.I.	P-Value
Salt Water	0.64	(0.33, 1.22)	0.175
Vegetable Oil	1.09	(0.57, 2.08)	0.792
Chamber (Ref. Ambient)			
Refrigerated Condition	1.63	(0.96, 2.79)	0.073
Day (Ref. Day 2)			
6	0.75	(0.36, 1.57)	0.439
10	1.02	(0.49, 2.14)	0.955
14	0.79	(0.37, 1.67)	0.54

**The odds ratio refers to the category associated with the odds ratio compared to the reference category*

Table 14: Categories of Failure Modes

Failure Type	Category
Peel	A Failure
Material Along Edge of Seal	B Failure
Peel + Material	C Failure
Delamination	D Failure
Material Away from Edge of Seal	E Failure

The second phase of the study requires a multiple comparison of means to analyze the effect of time, conditioning temperature, and contamination with seal strength at 140°C and 0.3 seconds sealing condition. The mean was calculated each test day for each contaminant over the 14 day period. A general linear model analysis compared the four recorded mean values for each contaminant shown in Table 15. The p-values for all factors for the FORCE (N) response and STRAIN (mm) response are greater than 0.05 ($\alpha = 0.05$). Therefore, we are 95% confident that there is no difference force (N) or strain (mm) when accounting for the effect of day, conditioning temperature, contaminant, day and contaminant interaction, day and chamber interaction, and chamber and contaminant interaction.

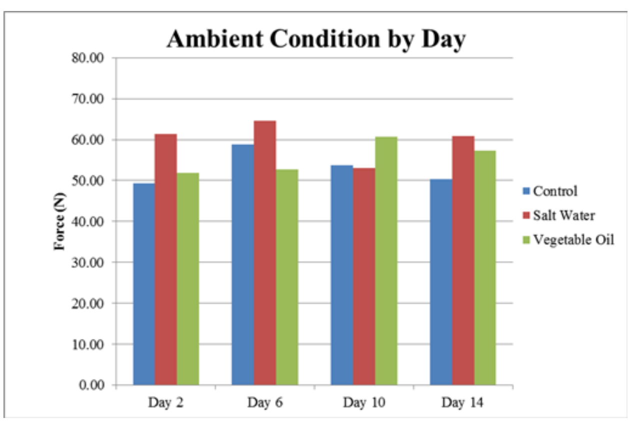

Figure 15: Seal Strength Results for Ambient Conditioning Temperature

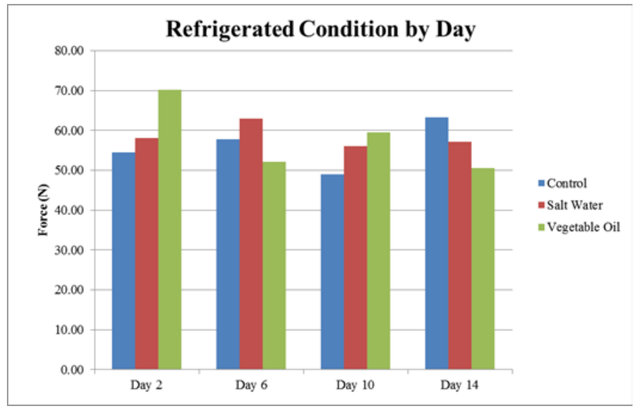

Figure 16: Seal Strength Results for Refrigerated Conditioning Temperature

3.3.3 Phase 2 Summary Findings

The statistical analysis indicates that there is no significant difference in Hermeticity for both contaminants and the control over the duration of Phase 2, nor is there a significant difference between contaminants and the control. In addition, there is no significant difference in seal strength between the contaminants and the control over the 14 day period; nor is there a significant difference in seal strength for each contaminant and the control throughout the 14 day period.

For the refrigerated conditioning temperature, vegetable oil has a similar Hermeticity pass rate to the control, and salt water has a lower pass rate across the 14 day test period. In addition, the ambient conditioning temperature shows vegetable oil with a higher pass rate for Days 2 and 6, but as stated previously there is no statistical difference. It should be noted that the sample size was less on day 14 for salt water and vegetable oil due to samples showing leaks at unwanted wrinkles or poor inflation during Hermeticity testing. The increase in inadequate inflation of the sample bags may be the result of gradual air loss over time. The sample bags may have had seal leaks, which allowed for enough air to release from inside the bag. Moreover,

Table 14: Categories of Failure Modes

ANOVA for Force (N)	
Variables	P-value
Day	0.863
Storage Temperature	0.606
Contaminant	0.372
Day*Contaminant	0.462
Day*Storage Temperature	0.67
Storage Temperature*Contaminant	0.742
ANOVA for STRAIN (mm)	
Variables	P-value
Day	0.949
Storage Temperature	0.499
Contaminant	0.525
Day*Contaminant	0.847
Day*Storage Temperature	0.615
Storage Temperature*Contaminant	0.629

the sample bags with inadequate inflation were discarded and not included in the represented data for Hermeticity. It was also observed that there was a visual difference in the aging of contamination over the 14 day period. The salt solution showed significant water loss from Day 2 to Day 14, which allowed for the salt and the red dye to be left behind. The vegetable oil did not show significant changes over the 14 days.

4.0 CONCLUSIONS

4.1 Significance of Contaminant Effect on Hermeticity and Seal Strength

The results of the study measured the effect of liquid contaminant at the T-point of the seal with a linear low-density polyethylene (LLDPE) sealant. Vegetable oil was used to simulate the effect of oil-based snack foods and the salt water solution was used to simulate salty snack foods. As mentioned previously, the T-point was chosen as the point of contamination because it is the most critical point of the seal. It was noted that the vegetable oil contaminant left residuals of oil onto the corrugated box during storage, which was determined in the study's findings that it may not necessarily be due to a failed hermetic seal. During production, the contamination was a continuous stream from bag to bag and may have been the reason for the oil found in the box. Therefore, the Hermeticity of the seal should be tested in addition to the visual observations made during quality inspection. Overall, it was determined that liquid contaminants found at the T-point do not have a significant effect on the Hermeticity or seal strength when using the study's method. Moreover, the effect of storage temperature and time do not have a significant effect on the performance of the LLDPE sealant when liquid contamination present in the seal area. Room temperature may be considered the more common storage temperature compared to refrigerated conditions, but it was important to determine if temperature was a factor to the performance of the sealant. The visual observation of the liquid contaminants between the two storage temperatures was seen to be the most different for the salt water solution. During the 14 day test cycle, the salt water solution progressed to a dry contaminant. Again, this can be due to the water vapor transmission rate of the film. Even though this study was used on a horizontal form, fill and seal, flow-wrap machine, the information can be useful for other snack food operating applications.

In addition to Hermeticity, the seal strength performance can be used to determine that the integrity of the seal strength of the T-point is not compromised with liquid contaminants. Each sample size included at least five sample bags with two replicates each at the front and back T-points. Therefore, the average of each sample bag was calculated to determine the average seal strength of the sample size. Overall, there was no significant difference between the test categories for the average maximum force experienced by the seal before failure. Prior to the study, it could have been assumed that the contaminants would lower the average seal strength to separate the film at the seal.

4.2 Significance of Sealing Temperature and Dwell Time

The different sealing temperatures and dwell time were chosen based on an acceptable range of sealing conditions and tested during Phase 1. The Phase 1 test was conducted to verify if different sealing conditions result in different hermetic seals. It would be more desirable to have a lower sealing temperature and dwell time for faster and lower production costs. Whereas, a high seal temperature and longer dwell time may be assumed to provide a higher Hermeticity pass rate. As it was observed that a sealing temperature of 160°C and 0.4 seconds dwell time had a higher pass rate than 140°C and 0.3 seconds dwell time. However, the binary logistic regression analysis indicates that the difference between the sealing temperatures and dwell times is not significant. In addition, 120°C sealing temperature and 0.2 seconds dwell time did have a significantly lower Hermeticity pass rate. The low seal initiation temperature of the LLDPE sealant allows for lower sealing conditions, but increasing the sealing temperature and dwell time will eventually plateau. The Hermeticity pass rate will eventually peak given the capacity of the molecular chain entanglement. Therefore, 140°C sealing temperature and 0.3 seconds dwell time is the optimal condition for producing a hermetic seal when accounting for vegetable oil and salt water liquid contamination for LLDPE sealant. Although not all sample bags had a hermetic seal, the purpose of the study was to compare the sealant layer's performance with no contamination to liquid contamination.

4.3 Significance of Results to Past Work

In comparison to some previous studies, practical and useful experimental conditions for the snack food industry were chosen for the test method. Some past work use dwell times greater than 0.5 seconds, which could lower the production efficiency if it was applied to industry. It may show better seal strength and Hermeticity trends on a large scale, but the scope of the study was to test a close range of sealing temperatures and dwell time that would most likely could be used in industry. Within the sealing conditions investigated, the optimal seal temperature and dwell time were different than the study conducted by Mihindukulasuriya and Lim [9] due to the wide range of dwell time and seal temperatures. However, Mihindukulasuriya and Lim [9] determined that vegetable oil has a slightly lower seal strength compared to water and the control. In addition to the findings of Mihindukulasuriya and Lim [9], this study uses statistical analysis to not only test for a difference between contaminants, but if the difference is significant. In addition, no work has been conducted for Hermeticity using a vacuum chamber, so the test method for the Hermeticity in this study can be used for future work.

4.4 Future Work

The suggested future work includes tested granular contaminants with the same method. The contamination system needs to be altered in order to apply a consistent stream of contamination. However, the same Hermeticity and seal strength methods can be applicable to other contaminants for flexible food packaging. It is also recommended to use twice as many samples for a stronger representation of the effect of liquid contamination on Hermeticity and seal strength. In addition, the frozen food industry can use this test method to investigate the effect of freezing conditions.

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